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Indian Standard

METHODS OF CHEMICAL ANALYSIS OF BRONZES

PART 3 DETERMINATION OF PHOSPHORUS – VOLUMETRIC METHOD

(First Revision)

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METHODS OF CHEMICAL ANALYSIS OF BRONZES

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METHODS OF CHEMICAL ANALYSIS OF BRONZES

PART 3 DETERMINATION OF PHOSPHORUS — VOLUMETRIC METHOD

(First Revision)

0. FOREWORD

0.1 This Indian Standard (Part 3) (First Revision) was adopted by the Bureau of Indian Standards on 22 July 1987, after the draft finalized by the Methods of Chemical Analysis of Non-Ferrous Metals Sectional Committee had been approved by the Structural and Metals Division Council.

0.2 IS : 4027 first published in 1967, covered determination of copper, lead, tin, manganese, phosphorus, nickel, iron, silicon, aluminium, zinc and antimony in bronzes. While reviewing this standard, the Sectional Committee decided that it is convenient to revise this standard in series of parts which, on publication will supersede the relevant method for determination given in IS : 4027-1967*. This part is one of that series and covers the determination of phosphorus by volumetric method. The other parts are as follows:

Part 1 Determination of copper and lead by electrolytic method

Part 2 Determination of manganese by photometric method

Part 4 Determination of nickel by photometric method

Part 5 Determination of tin by iodimetric method

Part 6 Determination of zinc by complexometric (EDTA) method

Methods for chemical analysis of other constituents in bronzes, namely, aluminium, iron, silicon and antimony are under preparation, and will be published as subsequent parts of above series.

0.3 In this revision, method for determination of phosphorus has been updated.

0.4 This method of analysis prescribed in this standard may primarily serve as referee method and may also be used by the laboratories for their day-to-day work. Due consideration has been given in the preparation of this standard to the facilities available in the country for such analysis.

*Methods of chemical analysis of bronzes.

0.5 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off it shall be done in accordance with IS : 2 - 1960*.

1. SCOPE

1.1 This standard (Part 3) prescribes a method for determination of phosphorus in the ranges as specified in the relevant Indian Standards on bronzes.

2. SAMPLING

2.1 Samples shall be drawn and prepared in accordance with IS : 1817 - 1961†.

3. QUALITY OF REAGENTS

3.1 Unless specified otherwise, analytical grade reagents and distilled water (*see* IS : 1070 - 1977‡) shall be employed in the test.

4. DETERMINATION OF PHOSPHORUS BY THE (ALKALIMETRIC) VOLUMETRIC METHOD

4.1 Outline of the Method — Phosphorus is precipitated as ammonium phosphomolybdate filtered, washed and dissolved in excess of sodium hydroxide. The unreacted alkali is titrated back against standard hydrochloric acid.

4.2 Reagents

4.2.1 *Concentrated Nitric Acid — rd* = 1·42 (conforming to IS : 264 - 1976§).

4.2.2 *Concentrated Hydrochloric Acid — rd* = 1·16 (conforming to IS : 265 - 1976‖).

4.2.3 *Ferric Chloride Solution* — Dissolve 2·5 g ferric chloride crystals ($FeCl_3.6H_2O$) in 400 ml of water and dilute to one litre, and add a few drops of concentrated hydrochloric acid.

4.2.4 *Ammonium Chloride* — Solid and 1 percent solution (*w/v*).

4.2.5 *Concentrated Ammonium Hydroxide* — 20 percent.

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*Rules for rounding off numerical values (*revised*).

†Methods of sampling non-ferrous metals for chemical analysis.

‡Specification for water for general laboratory use (*second revision*).

§Specification for nitric acid (*second revision*).

‖Specification for hydrochloric acid (*second revision*).

4.2.6 *Dilute Hydrochloric Acid* — 1 : 1 and 2 : 98 (*v/v*).

4.2.7 *Hydrogen Sulphide* — gas.

4.2.8 *Acidified Hydrogen Sulphide Solution* — Add 10 ml of concentrated hydrochloric acid to one litre of water and saturate with hydrogen sulphide.

4.2.9 *Ammonium Nitrate* — Solid.

4.2.10 *Ammonium Molybdate Solution* — Add solution A (**4.2.10.1**) to Solution B (**4.2.10.2**) with constant stirring, then add 0·1 g of ammonium phosphate dissolved in 10 ml of water and let stand for at least 24 h before using. Keep the solution in a cool place and always filter before using.

4.2.10.1 *Solution A* — Dissolve 100 g of molybdic acid (85 percent MoO_3) in a mixture of 150 ml of concentrated ammonium hydroxide and 270 ml of water.

4.2.10.2 *Solution B* — Mix 536 ml of concentrated nitric acid and 1 280 ml of water.

4.2.11 *Dilute Nitric Acid* — 2 : 98 (*v/v*).

4.2.12 *Potassium Nitrate Wash Solution* — Dissolve 10 g of potassium nitrate in water and dilute to one litre.

4.2.13 *Sodium Hydroxide Solution* — 0·10 N approximately.

4.2.14 *Phenolphthalein Indicator Solution* — Dissolve 0·5 g of phenolphthalein in 100 ml of 60 percent alcohol.

4.2.15 *Standard Hydrochloric Acid* — (0·1 N). Dilute about 11 ml of concentrated hydrochloric acid to one litre with water and standardize against standard sodium carbonate solution.

4.3 Procedure

4.3.1 Transfer 5·000 g of the sample into a 400-ml of breaker and dissolve in a mixture of 20 ml of concentrated nitric acid and 5 ml of concentrated hydrochloric acid. When dissolution is complete, add 10 ml ferric chloride solution and 5 g of ammonium chloride, dilute the solution to 200 ml, make ammoniacal, and heat to boiling. Filter and wash the beaker and the precipitate with hot ammonium chloride solution.

4.3.2 Transfer the paper and the precipitate to the original beaker, add 30 ml of dilute hydrochloric acid (1 : 1) and digest on a steam bath until the precipitate is completely dissolved. Bring the solution to boil, filter and wash ten times with hot dilute hydrochloric acid (2 : 98). Dilute the filtrate to 300 ml and pass hydrogen sulphide gas for 30 min. Allow the precipitated sulphide

to coagulate for two to three hours, and filter using a close texture paper. Wash the precipitate five times with acidified hydrogen sulphide solution. Mix the filtrate with that obtained in **4.3.1**.

4.3.3 Evaporate the combined filtrate to approximately 20 ml and transfer to 500-ml conical flask. Add 15 ml of concentrated nitric acid, boil gently for 10 minutes and add 100 ml of water. Cool the solution to 45°C and add 5 g of ammonium nitrate. Add 60 ml of the ammonium molybdate solution in small portions, while swirling the flask. Close the flask with a rubber stopper, shake vigorously for 5 minutes, and allow to stand for one hour. Filter, wash the flask and the precipitate three times with dilute nitric acid and then with potassium nitrate wash solution till 10 ml of the filtrate collected in a test tube does not consume more than one drop of sodium hydroxide solution using a drop of phenolphthalein indicator solution (begin testing the filtrate from third wash).

4.3.4 Transfer the paper and the precipitate to the flask, stopper the flask, add 30 ml of (carbon dioxide-free) water and shake vigorously until the paper is disintegrated. Wash down the stopper and sides of the flask, add 3 to 4 drops of phenolphthalein indicator solution, while shaking the flask, add sodium hydroxide solution from a burette until the pink colour persists and then add 2 ml in excess. Dilute to about 100 ml with water and titrate with standard hydrochloric acid until the pink colour is completely discharged.

4.3.5 Carry out a blank determination on the same quantity of sodium hydroxide solution which was added to the test, and titrate with standard hydrochloric acid in the presence of a few drops of phenolphthalein indicator solution.

4.4 Calculation

$$\text{Phosphorus, percent} = \frac{(A - B) \times C \times 0{\cdot}135}{D}$$

where

A = volume in ml of the standard hydrochloric acid required for the blank,

B = volume in ml of the standard hydrochloric acid required to titrate the excess sodium hydroxide,

C = normality of standard hydrochloric acid, and

D = mass in g of the sample taken.

BUREAU OF INDIAN STANDARDS

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